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# Sample Volume Effect on the Determination of Nitrate-Nitrogen in the Soil Profile

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## ABSTRACT

A field experiment was conducted to evaluate the effect and reliability of soil sample volume on nitrate-nitrogen ( $\text{NO}_3\text{-N}$ ) and soil-water content in the soil. Four soil sampling devices (a 20.3-cm power earth auger, a 5.1-cm hand earth auger, a 3.2-cm soil probe and a 1.9-cm soil probe) were used to collect soil samples from 35 conventionally tilled and 35 no-till locations. Soil samples were taken at 30-cm intervals to a depth of 150 cm with each of the four sampling devices and were analyzed for  $\text{NO}_3\text{-N}$  and moisture contents.

Results of this study indicate that sample volume has no significant effect on determining soil-moisture content. Sample volume also had no effect on  $\text{NO}_3\text{-N}$  concentration in the soil water under no-till conditions but had a definite impact on the  $\text{NO}_3\text{-N}$  levels in the conventionally tilled soils. Results further indicate that a 5.1-cm diameter sample may best predict the quantity of residual soil  $\text{NO}_3\text{-N}$ .

## INTRODUCTION

The possibility of residual nitrate-nitrogen ( $\text{NO}_3\text{-N}$ ) leaching into groundwater has been the center of much attention and concern. Many past investigators have suspected  $\text{NO}_3\text{-N}$  of leaching into groundwater systems and possibly causing adverse effects on the environment (Baker and Johnson, 1981; Galinato, 1987; Kanwar et al., 1982, 1985; Russelle et al., 1983). Other studies have shown that excess  $\text{NO}_3\text{-N}$  is very susceptible to leaching during periods of heavy rainfall or excessive irrigation (Adriano et al., 1972; Hooker et al., 1983). And more recent studies have found increased  $\text{NO}_3\text{-N}$  concentrations in tile drainage and groundwater as higher application rates of nitrogen fertilizer have been used (Kanwar et al., 1988; Schepers et al., 1986). Increased concentration of  $\text{NO}_3\text{-N}$  in tile effluents is an indicator of increased residual  $\text{NO}_3\text{-N}$  in the soil profile and shallow groundwater. This has led many observers to suspect a  $\text{NO}_3\text{-N}$  buildup in the soil caused by the overapplication of nitrogen fertilizer.

Rising energy costs and groundwater quality concerns

prodded many researchers and investigators in the Great Plains and Western states to develop programs to test soils for residual nitrogen (Ludwick et al., 1976, 1977; James, 1978). Many of the similar concerns and incentives exist today in the Midwest. But at present, there is no single standard in soil sampling for residual nitrogen. No one yet has developed a simple soil sampling program in the Midwest to determine the amount of residual  $\text{NO}_3\text{-N}$  available in the soil profile. Sampler type and volume are only some of the unanswered questions in developing such a program.

Augers and probes are both regularly used in soil sampling. Probes varied from the conventional hand-held sampling tubes (Ludwick et al., 1977; Malhi and Nyborg, 1983) to hydraulic rams (Linville and Smith, 1971; Schepers et al., 1986). Kanwar et al. (1985) combined the use of hand-held probes and hydraulic rams, whereas Hawley et al. (1982) simply used metal tubing. Adriano et al. (1972) employed a power-driven spiral auger and sampled to 15 m, while Hassen et al. (1983) used a bucket auger. Hassen et al. (1983) noted that the bucket auger inadvertently shaved the sides of the hole during sampling, possibly contaminating the sample.

Only a few studies have addressed the effect of sample volume on accuracy of  $\text{NO}_3\text{-N}$  available in the soil profile. Hassen et al. (1983) found that accuracy of available  $\text{NO}_3\text{-N}$  was affected by sample volume, and Peck (1983) found that as sample volume increased, variances in observation decreased. Hawley et al. (1982) found that a sample of 50  $\text{cm}^3$  or larger was required to provide a verifiable estimate, and samples for moisture content determination should weigh 200 g or more.

Many studies, previously mentioned, have given the diameters of the sampling devices used in their investigations. However, the diameter is dependent on length in order to provide an accurate sample volume. Hawley et al. (1982) recommended using 3.8-cm or larger-diameter probes for 10-cm-long cores for soil moisture measurement. This is 1.5 times or greater the diameter required to yield 50  $\text{cm}^3$  of sample from a 10-cm core, but it provides enough sample weight for soil-moisture determination. Considering all these restrictions, the minimum diameter for the 30-cm sample core suggested by James (1978) (assuming a bulk density of 1.4  $\text{g}/\text{cm}^3$  and a minimum final sample weight of 200 g) is 3 cm.

The objectives of this study were to determine the effect of the size of sample on  $\text{NO}_3\text{-N}$  and soil-water content and to determine the optimum sample volume for reliable determination of residual  $\text{NO}_3\text{-N}$  in the soil profile.

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TABLE 1. Physical Properties of the Soil for the Experimental Areas in Field Number 5 at the Agronomy and Agricultural Engineering Farm, Boone County, Iowa

Depth	No-till*				Conventional tillage			
	Particle size, $\mu\text{m}$			Bulk Density	Particle size, $\mu\text{m}$			Bulk Density
	sand 200-50	silt 50-2	clay 2		sand 200-50	silt 50-2	clay 2	
cm	%	%	%	g/cc	%	%	%	g/cc
0-30*	38.9	36.7	24.5	1.43	39.8	38.6	21.6	1.46
30-60*	35.5	37.8	26.8	1.33	41.3	36.5	22.2	1.45
60-90*	41.6	36.1	25.6	1.38	47.9	32.4	19.7	1.33
90-120*	53.1	25.2	21.7	1.44	55.1	29.6	15.3	1.45
120-150	-----	‡	-----	-----	54.8	31.0	41.2	1.65

\*Interpolated from Kanwar et al. (1987).

†From Galinato (1987).

‡Assumed to be that of conventional tillage.

## DESCRIPTION AND PROCEDURE

A field experiment, in which four different soil-sampling devices were used to evaluate the effect and reliability of sample volume on  $\text{NO}_3\text{-N}$  and soil moisture, was conducted at two sites at the Iowa State University Agronomy and Agricultural Engineering Research Farm west of Ames, Iowa. The experimental sites were selected for their known past management histories. The sites were part of plots that have been under their present tillage and fertility regime since 1984 (Kanwar et al., 1988). A single spring application (soon after planting) of 175 kg/ha (156 lb/ac) of nitrogen as 28% liquid has been applied to continuous corn in the conventionally tilled and no-tilled plots for the past four growing seasons (1984-1987).

Soils at the experimental site are from the Canisteo-Clarion-Nicollet Association formed under glacial till and are characterized by gentle rolling surface moraine with elevation differences normally less than 3 m (10 ft). This soil association makes up almost 80% of Boone County, Iowa soils. Surface drainage is poorly developed due to the undulating surface. Subsurface drainage is related to soil type (Canisteo soils are poorly drained, Clarion soils are well drained, and Nicollet soils are somewhat poorly drained), and permeability is moderate. Both test sites were principally Nicollet soils. Table 1 provides several physical soil properties for both conventionally tilled and no-till sites.

The four sampling devices comprised of a 20.3-cm (8 in.) power earth auger, a 5.1-cm (2 in.) hand earth auger, a 3.2-cm (1 1/4 in.) soil probe, and a 1.9-cm (3/4 in.) soil probe, were selected for their commercial availability and standardized sizes. The 20.3-cm diameter twist-style auger assembly included a detachable 107-cm (42 in.) flighted auger bit, a 90-cm (36 in.) flighted auger extension, and hydraulically actuated power head. The 5.1-cm diameter hand auger was a twist type with tee handle. Both the 1.9-cm and 3.2-cm diameter size soil probes were 45.7-cm (18 in.) long open-faced style sampling tubes with accompanying extension rods and tee handles.

Soil sampling was conducted over a 14-day period beginning 3 June and continuing through 16 June 1987. A small rain shower on the afternoon of 10 June interrupted sampling, but rainfall amount was not measurable and was intercepted entirely by the crop canopy. In each area, a five by seven grid layout was organized within 37 m (120 ft) by 54 m (175 ft) subareas. Both sites had 35 sampling locations (grid points)

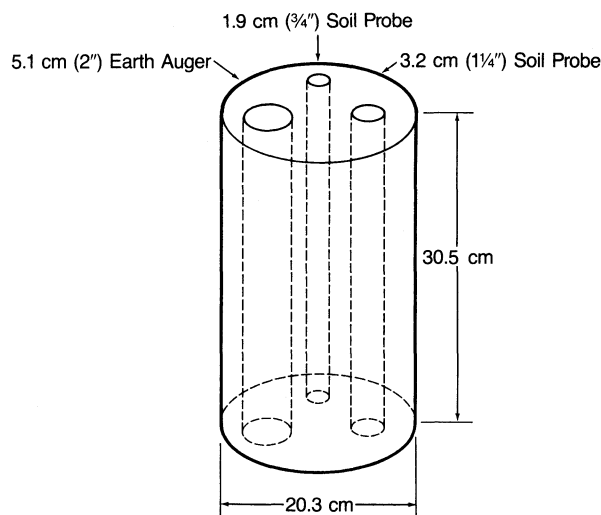


Fig. 1—Placement of smaller sampling devices within the 20.3-cm soil core.

approximately 7.6 m (25 ft) by 7.6 m (25 ft) from each other, forming the shape of equilateral parallelograms which were unique to each area.

Soil samples were taken to 150 cm (5 ft) depth with each of the four different-sized sampling devices at all locations. At the start of each location, a template was placed on the surface of the soil. Samples were then removed in 30 cm intervals. For each 20.3 cm diameter by 30-cm length profile of soil (Fig. 1), complete soil samples were removed first using the smaller 5.1-cm hand auger and then the 1.9-cm and 3.2-cm soil probes. This allowed for better placement of the bulkier 5.1-cm hand auger and more time to ready the auger for the next sampling layer. These samples were placed into prelabeled quart freeze bags and sealed. The remaining core was then removed with the power earth auger, thoroughly mixed, and subsampled for analysis. This subsample was also placed into a prelabeled bag and sealed. Each device was cleaned before continuing to the next depth.

The bagged soil samples, as they were collected, were placed into larger molecular weight poly bags by sampling location, then sealed and stored in large 30-gal polystyrene picnic coolers. This procedure was an attempt to reduce possible nitrogen and moisture loss during transport back to laboratory facilities for moisture and  $\text{NO}_3\text{-N}$  analysis. Soil samples were then stored for no more than 60 days at 1° C (34° F) in walk-in refrigerators until extraction were completed.

Extraction and analysis of the soil samples were performed by staff personnel of the analytical chemistry laboratory in the Department of Agricultural Engineering at Iowa State University. A 2 N potassium chloride (KC1) extraction procedure, as described by Keeney and Nelson (1982), was used to extract the  $\text{NO}_3\text{-N}$ . Soil moisture contents were determined gravimetrically. Sample extract solutions were analyzed for  $\text{NO}_3\text{-N}$  by the cadmium-reduction method.

## RESULTS AND DISCUSSION

Results from the laboratory analysis were used to determine means and variances for  $\text{NO}_3\text{-N}$

concentrations in soil water, volumetric moisture content, and residual soil NO<sub>3</sub>-N by individual depths for each of the four different-sized devices. A weighted average parameter was used to correct 20.3-cm auger values for statistical analysis in lieu of the 20.3-cm auger values obtained from the laboratory results because the smaller probes and auger samples were taken from within the 20.3-cm auger sample. The weighted average was used to reduce variability in the analysis which might have resulted from the possible removal or exclusion of high or low concentration areas from within the 20.3-cm sample by the smaller probes and auger. Weighted average values were calculated based on the relative volume for each of the four sampling devices as follows:

$$\text{Wt. avg.} = (A)0.008 + (B)0.0244 + (C)0.0625 + (D)0.9043$$

where

- A = 1.9-cm probe
- B = 3.2-cm probe
- C = 5.1-cm auger
- D = 20.3-cm auger.

It was assumed that the weighted average values represent the test sites' true means for NO<sub>3</sub>-N concentrations in soil water, volumetric moisture content, and the residual NO<sub>3</sub>-N.

Means and variances for NO<sub>3</sub>-N concentrations in soil water, soil moisture content, and residual soil NO<sub>3</sub>-N, along with the means and variances of their weighted averages, are listed in Tables 2 and 3 for the no-till and conventional tillage systems, respectively. The variance of NO<sub>3</sub>-N concentrations in soil water and residual soil NO<sub>3</sub>-N levels decreased with the increase in soil depth for all sample volumes under both tillage systems, whereas the variance of soil moisture content increased with the increase in soil depth. Also, a maximum level of

TABLE 3. Conventional Till Means and Variances of Soil Water NO<sub>3</sub>-N, Volumetric Moisture Content, and Residual Soil NO<sub>3</sub>-N  
n = 35

Depth cm	Device diam., cm	N(mg/l)		%M		N(kg/ha)	
		mean	vari	mean	vari	mean	vari
30	1.9	13.5	43.0	30.8	14.8	12.2	30.0
	3.2	19.4	189.4	29.7	8.4	17.0	132.0
	5.1	22.8	192.6	31.3	8.2	21.3	171.6
	20.3	18.9	112.6	29.7	9.9	16.5	78.4
	Wt. avg.	19.1	100.6	29.8	9.1	16.8	71.3
60	1.9	12.9	27.4	31.3	11.1	11.9	21.6
	3.2	14.4	45.6	31.8	17.1	13.6	40.6
	5.1	16.1	44.9	32.1	13.7	15.7	52.2
	20.3	16.5	67.2	32.1	10.3	15.7	48.6
	Wt. avg.	16.4	59.2	32.1	10.5	15.7	43.6
90	1.9	8.8	16.6	27.0	19.4	6.9	7.2
	3.2	8.2	7.9	27.4	18.4	6.5	3.8
	5.1	9.4	13.6	27.4	23.8	7.5	6.4
	20.3	11.4	9.1	26.8	16.6	9.0	4.6
	Wt. avg.	11.2	8.8	26.9	16.4	8.8	4.3
120	1.9	9.8	8.7	29.6	22.6	8.4	4.5
	3.2	10.5	15.0	30.2	22.8	9.0	8.4
	5.1	10.6	7.4	28.2	22.5	8.8	4.0
	20.3	11.5	8.3	30.4	25.1	10.4	6.1
	Wt. avg.	11.5	8.1	30.2	23.8	10.2	5.8
150	1.9	12.0	7.2	32.9	33.5	11.6	5.5
	3.2	11.8	5.9	34.9	34.9	12.2	6.0
	5.1	12.3	6.3	33.8	49.1	12.3	6.6
	20.3	12.2	5.3	36.5	33.4	13.2	5.7
	Wt. avg.	12.2	5.1	36.2	31.3	13.1	5.5

variability of residual soil NO<sub>3</sub>-N was in the top 60 cm of the soil profile. A comparison of data in Tables 2 and 3 clearly indicates that conventional tillage resulted in larger variations in the residual soil NO<sub>3</sub>-N in the top 60 cm of the soil profile when compared with the no-till practice.

Tables 4 and 5 give the amount of residual NO<sub>3</sub>-N in the soil profile as a function of tillage and sample diameter. On the average, more than 30% of the total profile NO<sub>3</sub>-N was found in the top 30 cm, and about

TABLE 2. No-till Means and Variances of Soil Water NO<sub>3</sub>-N, Volumetric Moisture Content, and Residual Soil NO<sub>3</sub>-N  
n = 35

Depth cm	Device diam., cm	N(mg/l)		%M		N(kg/ha)	
		mean	vari	mean	vari	mean	vari
30	1.9	18.7	77.5	31.6	11.3	17.6	67.1
	3.2	18.3	32.3	31.9	4.8	17.4	28.9
	5.1	24.6	99.1	31.3	18.3	22.3	58.9
	20.3	22.1	55.9	31.7	5.9	21.0	52.1
	Wt. avg.	22.1	50.7	31.7	5.7	21.0	46.8
60	1.9	13.5	43.7	30.7	5.4	12.4	36.6
	3.2	11.9	23.7	30.8	8.2	10.9	21.3
	5.1	16.4	90.5	29.7	7.9	14.5	69.5
	20.3	14.5	25.3	30.4	7.4	13.2	23.2
	Wt. avg.	14.5	24.5	30.4	6.9	13.2	22.2
90	1.9	10.9	16.0	27.0	12.0	8.7	9.7
	3.2	10.1	10.9	27.6	6.6	8.3	6.6
	5.1	10.8	12.9	27.3	13.2	8.8	8.0
	20.3	11.0	8.7	27.9	9.2	9.1	6.6
	Wt. avg.	10.9	8.5	27.9	8.9	9.3	6.3
120	1.9	9.7	18.4	29.3	25.4	8.2	7.3
	3.2	9.2	9.2	29.2	30.5	7.9	5.9
	5.1	9.8	9.4	27.4	19.7	8.0	6.5
	20.3	10.9	8.1	28.0	45.4	8.9	5.5
	Wt. avg.	10.8	7.2	28.0	40.0	8.8	5.4
150	1.9	10.2	7.5	33.9	64.5	10.1	6.1
	3.2	9.1	8.2	36.4	62.8	9.8	9.1
	5.1	10.7	10.6	33.3	63.0	10.3	7.8
	20.3	10.8	6.7	33.4	19.0	10.7	7.8
	Wt. avg.	10.7	6.2	33.5	17.8	10.7	7.5

TABLE 4. Average Residual Soil Nitrate-Nitrogen in the Profile as a Function of Sample Diameter and Tillage System

Device diam., cm	NO <sub>3</sub> -N (kg/ha)									
	1.0	3.2	5.1	20.3	Wt.Avg.					
Depth, cm	NT*	CT†	NT	CT	NT	CT	NT	CT	NT	CT
30	17.6	12.2	17.4	17.0	22.3	21.3	21.0	16.5	21.0	16.8
60	12.4	11.9	10.9	13.6	14.5	15.7	13.2	15.7	13.2	15.7
90	8.7	6.9	8.3	6.5	8.8	7.5	9.1	9.0	9.3	8.8
120	8.2	8.4	7.9	9.0	8.0	8.8	8.9	10.4	8.8	10.2
150	10.1	11.6	9.8	12.2	10.3	12.3	10.7	13.2	10.7	13.1
Total	57.0	51.0	54.3	58.3	63.9	65.6	62.9	64.8	63.0	64.6

\*NT = no-tillage.

†CT = conventional tillage.

TABLE 5. Percent of Cumulative Nitrate-Nitrogen in the Soil Profile as a Function of Sample Diameter and Tillage System

Device diam., cm	NO <sub>3</sub> -N (kg/ha)									
	1.0	3.2	5.1	20.3	Wt.Avg.					
Depth, cm	NT*	CT†	NT	CT	NT	CT	NT	CT	NT	CT
30	31	24	32	29	35	32	33	25	33	26
60	53	47	52	53	58	57	54	50	54	50
90	68	61	67	64	71	68	69	64	69	64
120	82	77	82	79	84	81	83	80	83	80
150	100	100	100	100	100	100	100	100	100	100

\*NT = no-tillage.

†CT = conventional tillage.

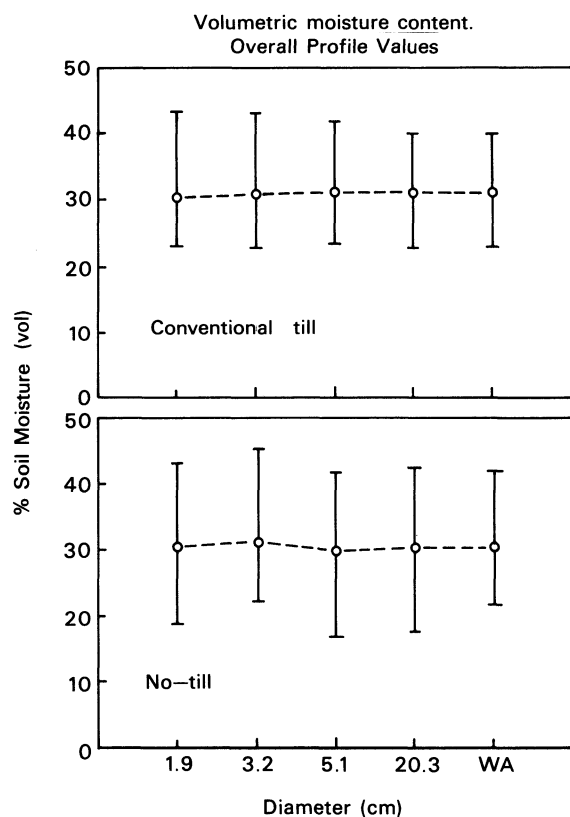


Fig. 2—Means and extreme values of profile volumetric soil moisture content for no-till and conventional tillages test areas.

80% of the total residual  $\text{NO}_3\text{-N}$  was found in the top 120 cm of the soil profile.

Statistical analysis for determining significant differences in means were performed by analysis of variance by using a 99% confidence interval. Critical value for  $F(0.01, 1, 68)$  in all the tests was 7.047. Analysis of variance testing of volumetric moisture content indicates that statistically, soil sample volume has no significant effect on moisture determination ( $F$ -values ranged from 0.02 to 3.87 and 0.00 to 6.00 for no-till and conventional tillage, respectively). Because soil moisture content is used in the determination of both soil-water  $\text{NO}_3\text{-N}$  concentration during laboratory analysis and cumulative residual soil  $\text{NO}_3\text{-N}$  content, differences in soil moisture content attributed to sample volume error can jeopardize accuracy in calculating these two values. Soil moisture profile means show little difference between soil moisture content in the top 150 cm soil profile for both tillages (Fig. 2).

Analysis of  $\text{NO}_3\text{-N}$  concentrations in soil water (in mg/l) from the no-till plots failed to display any significant differences in sampling volumes by individual depth.  $\text{NO}_3\text{-N}$  concentrations in soil water in conventional tillage plots failed to accept the 1.9-cm probe for 30-cm and 90-cm depths ( $F = 7.60$  for both depths) and also the 3.2-cm probe at the 90-cm depth ( $F = 18.95$ ). Cumulative residual soil  $\text{NO}_3\text{-N}$  (profile totals or the aggregated total of all five depths in kg/ha) were used to evaluate residual soil  $\text{NO}_3\text{-N}$  because corn plants utilize  $\text{NO}_3\text{-N}$  from the entire root zone profile (approximately 150 cm). The cumulative  $\text{NO}_3\text{-N}$  total for the 3.2-cm probe under no-till conditions was found to

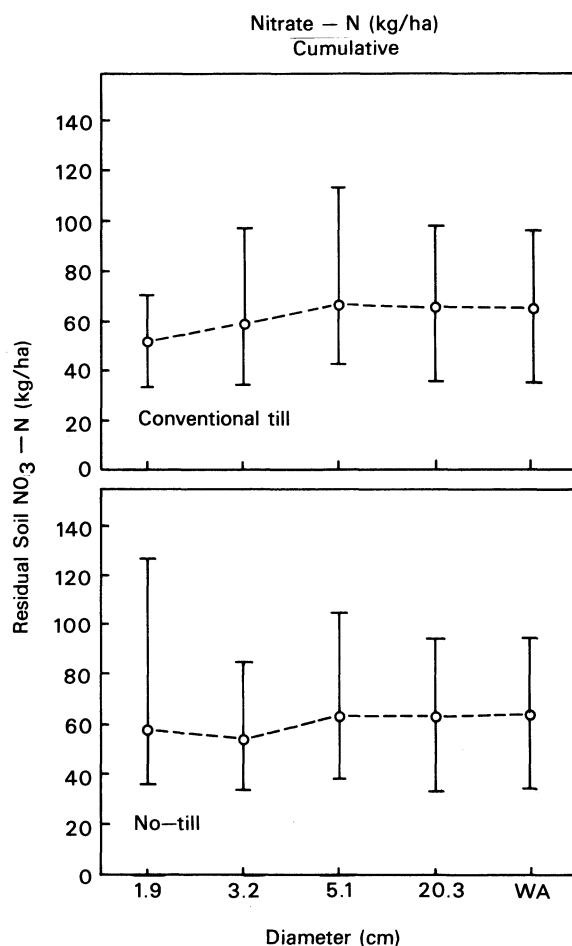


Fig. 3—Means and extreme values of cumulative residual soil  $\text{NO}_3\text{-N}$  for no-till and conventional tillages test areas.

be significantly different from the weighted average value ( $F = 7.19$ ). Analysis of conventional tillage conditions rejected the 1.9-cm probe ( $F = 12.87$ ) for use in measuring cumulative residual soil  $\text{NO}_3\text{-N}$ . Figure 3 shows cumulative residual soil  $\text{NO}_3\text{-N}$  trends for the several sample volumes and reflects some similarities between the different tillages, especially for larger volumes. The 1.9- and 3.2-cm probes seem to have lower mean values from the larger volumes. Cumulative profile totals of residual  $\text{NO}_3\text{-N}$  for the 5.1-cm auger were not significantly different from the weighted average means. Thus, the sample volumes provided by the 5.1-cm auger may best predict cumulative profile nitrate-nitrogen quantities.

## SUMMARY

A total of 1400 soil samples were taken from no-till and conventional tillage plots during the first two weeks of June 1987 for  $\text{NO}_3\text{-N}$  and soil-water analysis. Four different-sized sampling devices were used to collect soil samples from 35 locations to a depth of 150 cm under each tillage system. Soil samples were then extracted and analyzed for soil water  $\text{NO}_3\text{-N}$  concentration and soil-moisture content in the analytical chemistry laboratory of the Department of Agricultural Engineering at Iowa State University. The results of this study support the following conclusions:

1. Sample volume had no clear effect on the determination of moisture content of soils. None of the three sample volumes tested gave values significantly different from the overall weighted average value. In addition, little difference was seen between no-till and conventional tillage soil moisture contents for the entire 150-cm soil profile.
2. Sample volume did not effect the determination of  $\text{NO}_3\text{-N}$  concentrations in the soil water for no-till but did effect the determinations of  $\text{NO}_3\text{-N}$  concentrations in soil water for the conventionally tilled soils in this study.
3. The sample volume provided by the 5.1-cm auger may best predict the cumulative quantity of residual soil  $\text{NO}_3\text{-N}$ . Analysis of cumulative soil  $\text{NO}_3\text{-N}$  showed that, statistically, there was no difference between the sample volume of the 5.1-cm auger and weighted average test volume values for either tillage types. However, acquisition of soil samples by the 5.1-cm auger may prove too demanding and exhausting to be practical. Therefore, substitution of a 5.1-cm probe may prove a more practical choice.

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